

UV/EB Chemistry and Technology

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The dominant chemistry for UV and EB curing is the free radical polymerization of unsaturated monomers and oligomers. Monomers and oligomers containing acrylate unsaturation are the most commonly utilized in UV/EB-initiated free radical polymerization due to their higher reactivity compared to methacrylate, allyl or vinyl unsaturation.¹ (See Figure 1.)

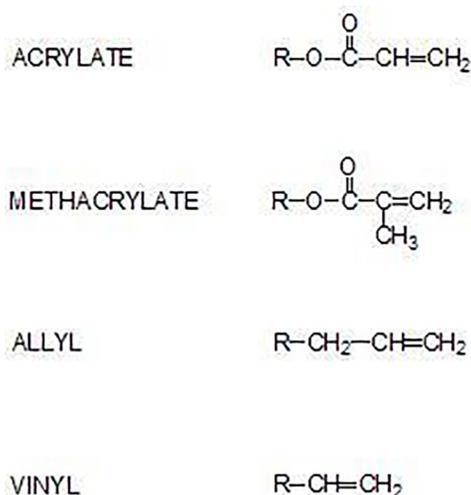


FIGURE 1. Types of unsaturation

While specific formulations will vary greatly depending on end-use requirements, the majority will contain one or more of the following components:

1. **Multifunctional acrylate oligomers**, which impart the basic properties of the cured ink or coating.
2. **Multifunctional acrylate monomers**, which have the major function of providing lower ink or coating viscosity but they also can be used to modify film properties, such as hardness, adhesion and chemical resistance.
3. **Monofunctional acrylate monomers**, which lower ink or coating viscosity and reduce cross-linking in the cured film. Volumetric shrinkage is reduced for improved adhesion, and flexibility and impact resistance are increased.
4. **Photoinitiators**, which absorb energy and initiate polymerization (not necessary in EB free radical polymerization).
5. **Additives and pigments**, which are similar to those used in conventional cure and will not be discussed in this paper.

Acrylate oligomers

The acrylate oligomers used in UV/EB radical polymerization are typically viscous liquids ranging from a few thousand to

Quick summary

Free radical polymerization of acrylate monomers and oligomers is the most popular chemistry used in the UV/EB curing of inks and coatings.

The chemical structure of the monomers and oligomers determines the physical properties of a formulation before and after curing.

By understanding the chemical structures of the materials used in UV/EB curing, one can predict viscosity, reactivity, chemical resistance, flexibility, weathering, adhesion and cost.

Oxygen inhibition of free radical polymerization exists, and it must be considered when designing formulations and curing systems.

Although the free radical curing mechanism is the same for UV and EB, EB uses no photoinitiator, and it is inerted with nitrogen to mitigate oxygen inhibition and to prevent ozone formation.

UV/EB cationic cure systems shrink less than free radical cure systems, thus they have better adhesion, especially on unprimed metals and plastics.

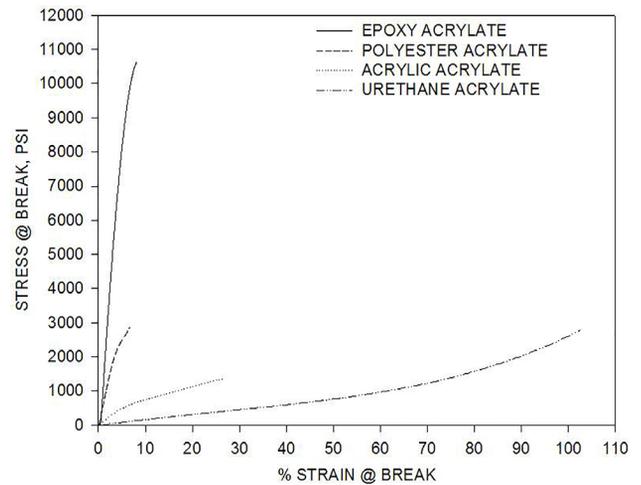
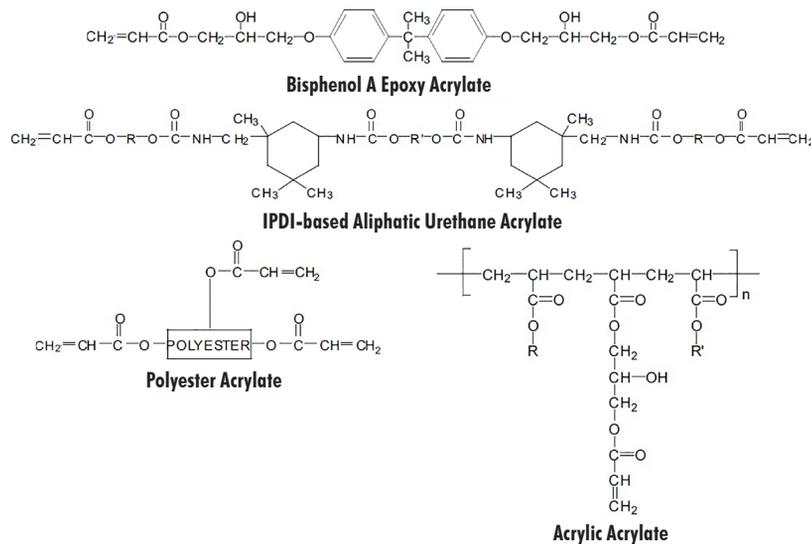
greater than 1 million centipoise in viscosity at 25°C. They also typically possess two to six acrylate groups per molecule and range in molecular weight from approximately 500 to 20,000.

The acrylate oligomers provide film properties superior to what can be achieved with monomers alone. Table 1 lists several types of acrylate oligomers commonly used in UV- and EB-curable inks and coatings along with their typical effect and use in ink/coating performance. Combinations of these oligomer types often are used to optimize the coating performance. Figure 2 provides some typical structures of acrylate oligomers.

This variety of oligomer types yields UV- and EB-cured polymers with a wide range of physical properties. There are oligomers that form hard, rigid polymers with high tensile strength and modulus, and others that form soft, extensible polymers with high elongation. Graph 1 displays stress/strain curves of UV-cured polymers typical of each oligomer type, and illustrates the variety of properties that are possible.

Oligomer Type	Performance Effects	Use in Inks and Coatings
Epoxy acrylate	Increase reactivity, hardness, chemical resistance; decrease cost	Oligomer of choice for coatings; used to lower cost in inks
Aliphatic urethane acrylate	Increase flexibility, toughness, weathering; multi-functionals increase reactivity, hardness, chemical resistance; decrease yellowing	Increase flexibility or hardness; for touch, weatherable screen inks
Aromatic urethane acrylate	Increase flexibility, toughness; multifunctionals increase reactivity, hardness, chemical resistance; decrease cost (vs. aliphatic)	Increase flexibility or hardness; not weatherable
Polyester acrylate	Increase wetting; decrease viscosity	For pigment wetting, adhesion; oligomer of choice for litho inks due to water balance and printability
Acrylic acrylate	Increase adhesion, weathering	Increase adhesion

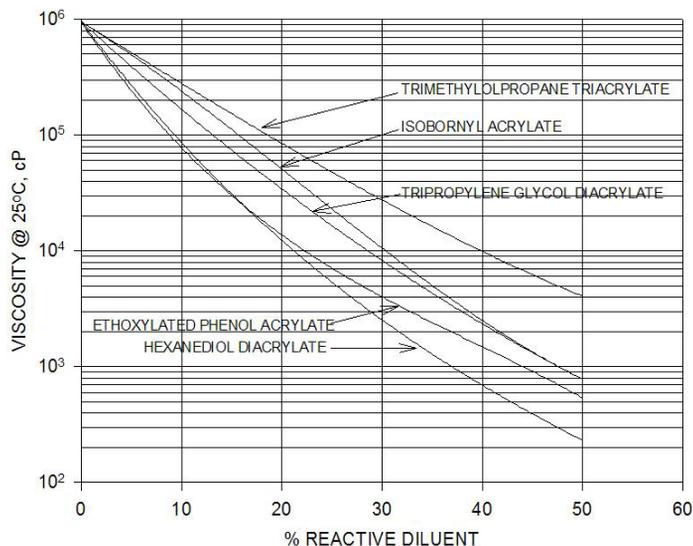
TABLE 1. Acrylate oligomer types, their performance effects and use in inks and coatings



GRAPH 1. Typical tensile properties of acrylate oligomer types*

* It should be noted that within each oligomer type, there exists a variety of structure variations to modify physical properties. These variations can result in epoxy acrylates with high elongation and urethane acrylates with high tensile strength and modulus.

FIGURE 2. Typical structures of acrylate oligomers



GRAPH 2. Viscosity reduction of selected monomers

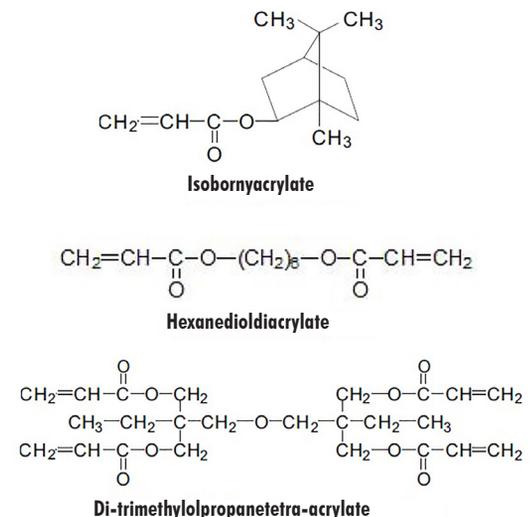


FIGURE 3. Structures of typical monomers

Acrylate monomers

The acrylate monomers used in UV and EB curing typically have one to four acrylate groups and range in molecular weight from approximately 150 to 500. The monomers are commonly light-colored liquids with viscosities from 5 to 200 centipoise at 25°C. Volatility ranges from low to moderate.

Acrylate monomers act as diluents for the acrylate oligomers in order to achieve ink and coating viscosities suitable for typical application equipment. Graph 2 demonstrates the viscosity reduction of several common monomers on a high viscosity acrylated oligomer. In this case, hexanedioldiacrylate is the best diluent.

Unlike solvents, the acrylate monomers co-polymerize with the acrylate oligomers to form an integral part of the cured ink or coating and can have significant effects on the coating performance.

The functionality of the monomer has a very significant effect on properties of the cured coating. Table 2 lists the typical performance effects of each monomer type. Figure 3 shows the structures of typical monomers.

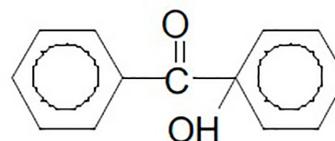
Like the acrylate oligomers, the acrylate monomers within each type exhibit a considerable range of performance. Some monofunctional acrylate monomers will impart greater hardness to a coating than others, while some trifunctional acrylate monomers provide more flexibility relative to other triacrylate monomers. Mixtures of monomers typically are used to optimize the performance of a formulation.

Photoinitiators

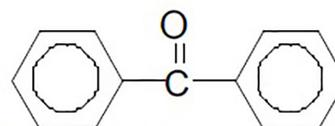
Photoinitiators are chemicals that form energetic radical species when exposed to UV light. They are essential ingredients in UV-curable inks and coatings, necessary in order to obtain polymerization. Depending on such factors as film thickness, UV light source and particular ink or coating performance requirements, the amount of photoinitiator in a

UV ink or coating formulation can range from approximately 0.5% to 15%. Photoinitiator systems vary widely in composition depending on the particular requirements needed: very thin ($\leq 6\mu$) clear coatings vs. thin ($\leq 25\mu$) pigmented inks or coatings vs. very thick ($\geq 100\mu$) clear coatings. Mixtures of photoinitiators often are used to ensure both surface and through cure of an ink or coating.

There are two main types of free radical photoinitiators, Type I and Type II.² Figure 4 shows the structures of typically used Type I and Type II photoinitiators. Type I photoinitiators are those compounds that undergo cleavage upon irradiation to generate two free radicals. Generally, only one of these free radicals is reactive and goes on to initiate polymerization. The non-reactive free radical contributes to migrating species and extractables, causing problems in food packaging applications. Benzoin and benzoin ethers were some of the earliest used Type I photoinitiators. Today, 1-hydroxy-cyclohexylphenyl-ketone is widely used.



Type I. 1-hydroxy-cyclohexylphenyl-ketone



Type II. Benzophenone

FIGURE 4. Types of photoinitiators

Type II photoinitiator systems are those compounds that form an excited state upon irradiation and then abstract an atom or electron from a donor molecule (synergist). The donor molecule then acts as the initiating species for polymerization. A widely

Monomer Type	General Performance Effects	Comments
Monofunctional	Reduce cross-linking, lower shrinkage, increase adhesion, best viscosity reduction, can increase residual uncured material; decrease chemical resistance and reactivity	May cause swelling of photopolymer printing plates and rollers (not used in litho); used extensively in screen for adhesion and flexibility
Difunctional	Good compromise between flexibility, hardness, viscosity reduction, chemical resistance and reactivity	Aggressive monomers may cause swelling of photopolymer printing plates and rollers (hexanediol diacrylate not used in litho)
Trifunctional and greater	Increase cross-linking, reactivity, hardness, chemical resistance, scratch resistance, shrinkage; decrease flexibility and adhesion; not as effective in reducing viscosity	Some are good pigment wetters (propoxylated glycerol triacrylate); ethoxylated monomers can improve flow

TABLE 2. Acrylate monomer types and performance effects

used Type II photoinitiator system contains benzophenone. Tertiary amines typically are used as synergists because they react with benzophenone very efficiently, and they also serve to retard the inhibition of polymerization by oxygen. Acrylated tertiary amine compounds are used when odor and extractables are of concern. Benzophenone, which has a distinctive odor, is excluded from many food packaging applications.

Polymeric and oligomeric photoinitiators have been developed to limit migration and extraction in food packaging applications. However, solubility and reactivity can be negatively impacted by these types of photoinitiators.

In EB curing, the core ingredients of the coating formulations are the same as in UV curing. However, the addition of the photoinitiator is not necessary. The electrons generated by the EB-curing equipment carry sufficient energy to lead to initiation of the polymerization process. This makes EB cure more acceptable for food packaging than UV cure.

Free radical polymerization

The free radical UV polymerization process is shown in Figure 5.³ In this figure, the photoinitiator, I, absorbs light and forms a radical species. This radical species then reacts with an acrylate functional material, R, in the initiation step. Propagation occurs as the reactive species or growing polymer, IR^{*}, reacts with additional acrylate functional materials. The propagation step is the process that builds the molecular weight and the cross-linking of the polymer. Propagation continues for as long as the reactive species can locate an acrylate functional material or until chain transfer or termination occurs. At some point in the process, the mobility of the growing polymer, IR^{*}, will be limited by the viscosity build of the system (caused by the increase in molecular weight and cross-linking of the polymer). This lack of mobility will limit its ability to find acrylate functional materials, and the propagation reaction will stop. Chain transfer occurs when the growing polymer abstracts a hydrogen atom from a donor molecule. This chain transfer stops the polymerization of the growing polymer and begins the formation of a new polymer. Thus, chain transfer can be used to limit the molecular weight of a polymer. Termination occurs when two growing polymer chains react with each other to stop polymerization.

In EB free radical polymerization, there is no specific radical formation step as there is in UV since photoinitiators are not used. The formation of initiating radicals usually involves the interaction of the electrons onto the acrylate functional materials themselves, with the resulting formation of the reactive species, R^{*}. The reactive species then reacts via the same propagation, chain transfer and termination steps as with UV.

Oxygen inhibition

Figure 6 shows the reactions of oxygen with photoinitiator (PI)

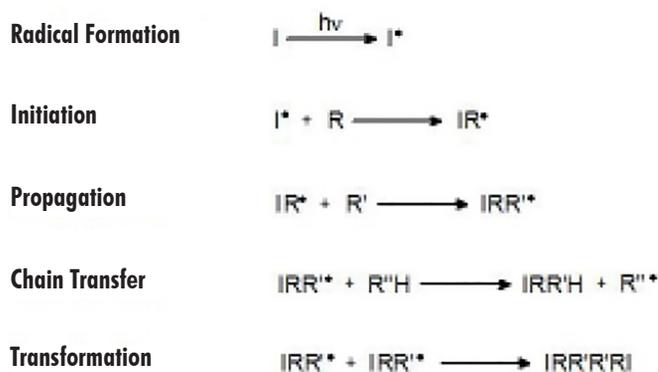


FIGURE 5. UV free radical polymerization process

QUENCHING REACTION



SCAVENGING REACTION

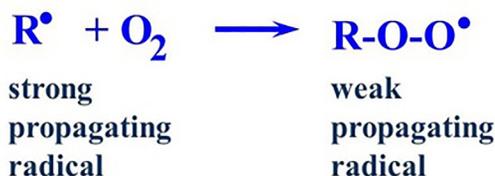


FIGURE 6. Reactions of oxygen in UV-initiated free radical polymerization

and free radicals (R^{*}). In the reaction of oxygen with the excited state of the photoinitiator, the photoinitiator is quenched and returns to an unexcited, non-reactive state. The end result is fewer free radicals produced by the photoinitiator, with fewer initiated polymer chains, and less polymer formation in the coating. When the triplet state of oxygen reacts with a carbon-based free radical, it converts the growing chain to an oxygen based free radical. This oxygen-based radical is less reactive than the carbon-based radical, and it slows down the free radical polymerization, resulting in lower molecular weight chains. In either case, the reaction with oxygen may give a range of results, from reduced coating properties to uncured, liquid surfaces on the coating.

Since oxygen is present at about 21% in air, oxygen inhibition of free radical polymerization is a very real problem in the energy cure industry.⁴ Oxygen is present in two places: in the bulk material and at the surface of the coating. Oxygen is generally required in the bulk material to provide storage stability. (In this case, the oxygen inhibition is a welcome attribute.) This bulk oxygen is consumed fairly rapidly upon exposure to UV and has little impact for most coatings. The oxygen at the coating surface is replenished as it is consumed and causes most of the issues seen in the industry. Since this

oxygen is at the surface of the coating, oxygen inhibition frequently is referred to as a surface cure problem.

There are known physical and chemical ways to reduce oxygen inhibition or improve surface cure:

1. Remove oxygen from the UV cure zone by use of inert gas, by use of waxes that migrate to the surface and form a barrier, or by use of films that are in direct contact with the coating.⁵ In UV waterbased systems, the water vapor may act as an inert gas.⁶
2. Increase free radical concentration by increasing the photoinitiator concentration or the light intensity (irradiance).⁵
3. Use chemicals that react with the peroxy radicals.⁵ These solutions have been and are being used in the industry, but they all have advantages and disadvantages.

The removal of oxygen is not an easily implemented or cost-effective solution. Nitrogen inerting is used in some specialty applications, but the typical end user cannot afford the cost. Inerting a web-based application also can be especially difficult. Waxes can be used, but they impact the final properties of the coating, and time is needed for migration of the waxes to the surface of the coating. Laminating adhesives is an example of an application that uses film as an oxygen barrier. In this case, the film becomes part of the product. When water is evaporated from waterbased UV coatings, the water vapor acts as a barrier to oxygen. If UV cure is immediate after water evaporation, very good surface cure is obtained.⁶

The light intensity is determined by the available UV-curing equipment and often cannot be changed. Higher intensity lamps are also generally more expensive. Increased photoinitiator levels may result in increased concentrations of undesired residuals or by-products. It also may result in lower molecular weight polymer chains and reduced coating properties, due to increases in initiation and termination reactions. (See Figure 5.)

The use of reactive chemicals is the most commonly implemented solution to mitigate oxygen inhibition. The chemicals that react with peroxy radicals contain easily abstractable hydrogen atoms. These abstractable hydrogen atoms are found in compounds containing specific structural elements, sulfur (thiols), nitrogen (amines) or oxygen (ethers). The hydrogens on the carbon atoms alpha to the sulfur, nitrogen or oxygen are those that are easily abstractable, and there are many of these in each molecule. The efficacy of these compounds, for the same equivalency, is thiols > amines > ethers. Improvements in performance are noted when the thiols, amines and polyethers are acrylated. The acrylate functionality ensures the materials become part of the polymer backbone and cannot migrate or bloom to the surface. A reduction in odor also may be obtained through acrylation.

The dominant chemistry for UV and EB curing is the free radical polymerization of unsaturated monomers and oligomers.

One drawback of the use of thiols is odor. However, improvements in purity have reduced the odor of many thiols. Advantages of using thiols may be improved thermal resistance, reduced moisture absorption and improved adhesion. The use of amines can result in yellowing (either upon cure or after cure), residual odor and moisture sensitivity. The yellowing can be masked or lessened through use of dyes or optical brighteners. Ethers are the least effective solution to oxygen inhibition but can be used in large quantities via polyether structures. Many urethane acrylates utilize polyether backbones based on polyethylene oxide, polypropylene oxide or polybutylene oxide. Since large amounts of these polyethers are required, the performance properties of the coating are affected by polyether choice. Diluting acrylates also may be ethoxylated or propoxylated to give reduced oxygen inhibition. The ether groups can cause reduced temperature resistance of coatings, and depending on the type of polyether, reduced water resistance.

The polyethers may be used alone or in combination with the thiols or amines to provide further improvements in cure performance. When polyethers are used alone, the deficiencies of the thiols (odor) and amines (yellowing, odor, moisture sensitivity) are absent. The ultimate cure speed of these products is determined by the molecular weight, the acrylate functionality, the type and amount of modification, and the type of resin (epoxy acrylate, urethane acrylate, polyester acrylate, etc.). See Table 3 for a summary of the methods to mitigate oxygen inhibition, as well as the advantages and disadvantages of each.

FORMULATION AND CURE BASICS

Better surface cure also can be improved via conventional formulating guidelines and design of cure equipment. Increasing the functionality or double bond concentration of the coating gives a more cross-linked or harder coating. There are also certain structures that provide for highly reactive materials.⁷ Increasing the viscosity of the coating decreases the oxygen diffusion into the coating and improves the surface cure.⁵ Thicker coatings suffer less from oxygen inhibition due to the bulk polymerization reaction, which increases viscosity and significantly reduces oxygen diffusion.⁸ Decreasing the distance from the lamp to the substrate increases the irradiance delivered to the substrate. Increasing the exposure time, via

Method	Advantages	Disadvantages
Inert Gas	Does not adversely affect coating properties	Expensive; difficult to implement
Waxes	Inexpensive	Affects final coating properties; time needed for migration
Films	Good solution when film becomes part of project	Cost/disposal of film when not part of product
Increase PI concentration	Easy to implement	Increased residuals/by-products; reduced coating properties
Increase light intensity	May not affect coating properties	Part of existing equipment; cost
Reactive Chemicals		
Thiols	Improved thermal resistance; reduced moisture absorption; improved adhesion	Odor
Amines	Inexpensive; possible improved adhesion	Yellowing upon or after cure; residual odor; moisture sensitivity
Ethers	Can be used in large quantities	Affects coating properties; reduced temperature resistance; possible reduced water resistance
Structural Elements	Can provide good coating properties	Availability; cost; undesired properties

TABLE 3. Methods to mitigate oxygen inhibition, with advantages and disadvantages

slower cure speeds or multiple lamps, also generally increases the extent of cure. These basics can be combined with the oxygen inhibition mitigation techniques to further improve coating performance.

Cross-linking of polymers

Cross-link density is an indication of how tightly a polymer network is interconnected, and it is predictive of many of the properties of the final ink or coating.⁹ The weight per acrylate double bond of the formulation can be used to provide an indication of the cross-link density. To obtain the weight per acrylate double bond, divide the average molecular weight of the formulation by the average functionality of the formulation. The most common method of reducing cross-link density is through the addition of monofunctional monomers to the formulation. Table 4 lists some properties that are affected by cross-link density or weight per double bond.

Low	Weight per double bond	High
Property		
Faster	Cure Speed	Slower
Lower	Flexibility	Higher
Higher	Tensile Strength	Lower
Higher	Shrinkage	Lower
Higher	Chemical Resistance	Lower
Worse	Adhesion	Better

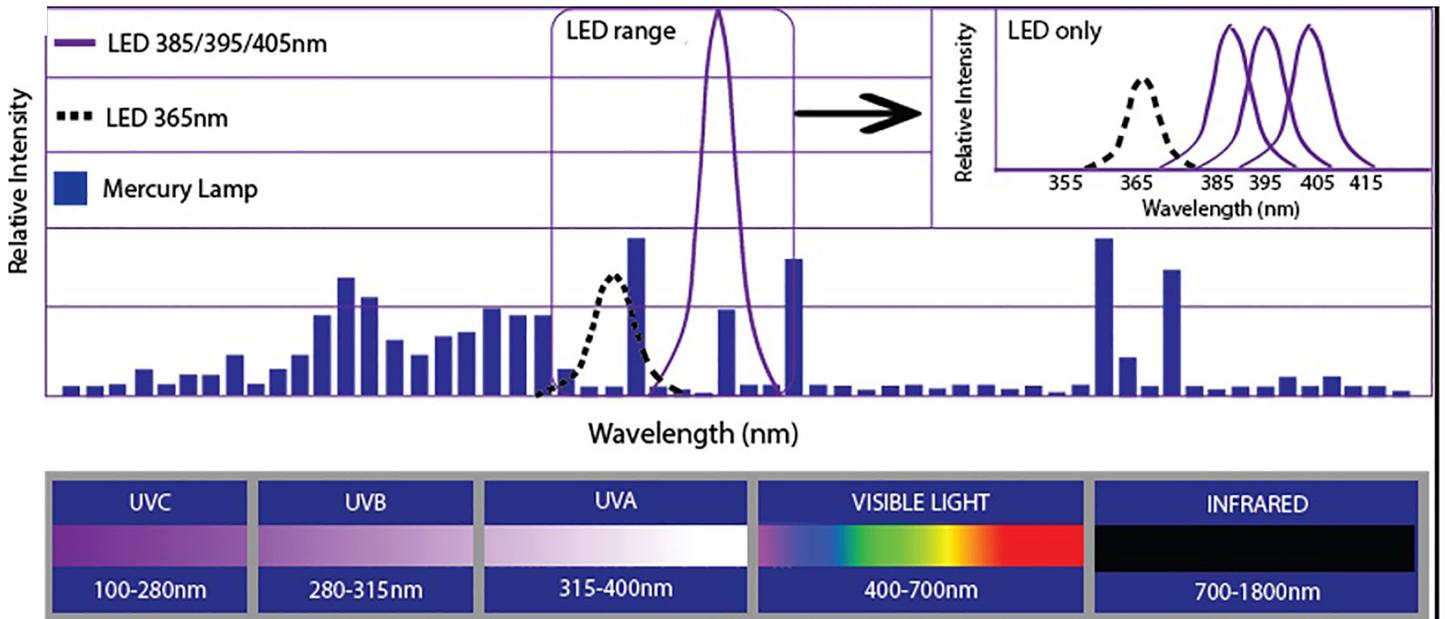
TABLE 4. Effect of weight per double bond on properties of inks and coatings

UV lamp types

UV curing can use many different types of lamps. The earliest and most widely used is the full-spectrum mercury (Hg) lamp. It emits energy over a wide range of wavelengths, from the UVC to the IR. (See Figure 7.) The mercury lamp can be doped with various elements to modify the wavelength emission. Figure 8 shows the emissions of several types of doped lamps. Filters can be used to limit the range of wavelengths that are emitted. Short wavelengths may be filtered to limit ozone production and the need to remove it with ventilation controls. Long wavelengths may be filtered to limit heat generation, allowing a broader range of substrate use.

UV-LEDs are a recently introduced lamp type. Compared to mercury lamps, they are seen as more energy-efficient, more environmentally friendly (no ozone or mercury), more substrate-tolerant (lower cure temperature) and longer lived (less maintenance). The UV-LED lamp emissions are mostly monochromatic and in the UVA wavelength range (385-405nm). (See Figure 7.)

Some of the newer installed UV cure systems deliver less energy to the coating and eliminate shorter wavelength UV. Both of these factors tend to increase the impact of oxygen inhibition on UV cure. Increasing the irradiance increases the concentration of free radicals, and higher concentrations of free radicals consume more oxygen. Conversely, low energy cure systems, with lower irradiance, result in lower concentrations of free radicals and more oxygen inhibition. The irradiance



*courtesy of Phoseon Technology

FIGURE 7. Mercury lamp and LED emission spectra

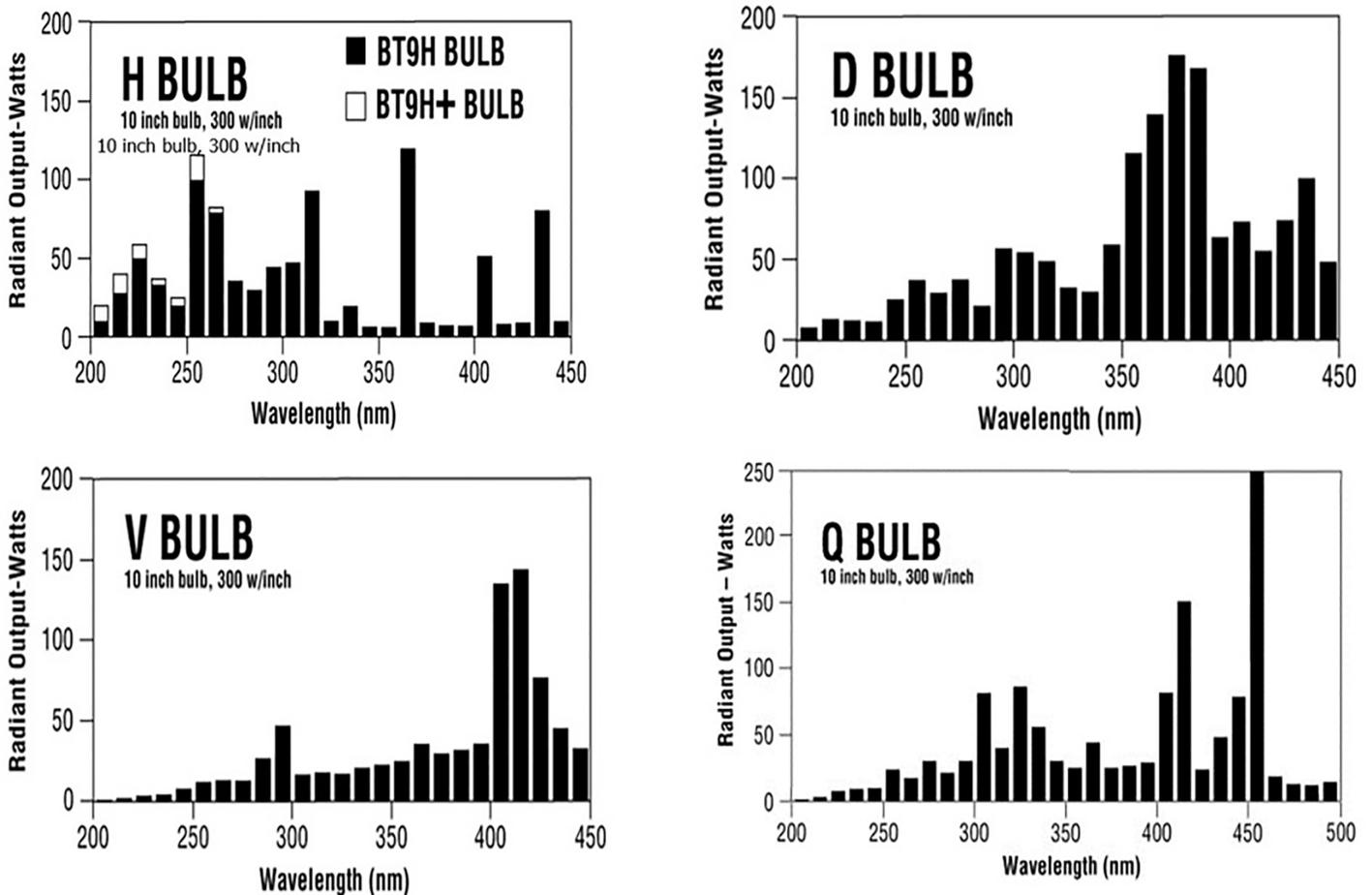


FIGURE 8. Spectral output with different types of bulbs

should be measured at the substrate to determine the delivered energy because delivered energy decreases with distance from the lamp to the substrate.

There is a wavelength dependence on absorption of UV. Shorter wavelengths (UVC) tend to be absorbed at the surface of a coating, while longer wavelengths (UVA) tend to penetrate the coating to be absorbed near the substrate, and mid-wavelengths (UVB) are absorbed near the middle of the coating. (See Figure 7 for wavelength nomenclature.) Some of the newer energy cure systems do not emit UVC wavelengths, resulting in increased oxygen inhibition at the surface of the coating.

UVA lamps, by definition, primarily emit wavelengths in the UVA range. Most LED lamps emit at 395nm, also in the UVA range. LED lamps with 385nm and 405nm emissions are also used, and lamps with 365nm emission are available but at lower intensity. (See Figure 7.) The LED lamps have seen a steady increase in peak irradiance, from 1.1 watts/cm² to 20 watts/cm². Improvements in the optics of UV-LEDs also have led to an increase in the irradiance that is delivered to the substrate surface.

Comparison of UV and EB curing

The UV and EB processes share some key performance characteristics. Both are efficient compared to thermal curing methods, consuming many times less energy. Neither process exposes a substrate to high temperatures, allowing a high degree of cross-linking of inks and coatings on temperature sensitive material. Both UV- and EB-curing equipment require only a few square meters of installation space. Of greatest significance, both processes can convert low viscosity liquids to durable polymer inks or coatings nearly instantaneously.

Very significant distinctions exist between the UV- and EB-curing processes. The free radical polymerization common to both processes is inhibited by oxygen in the atmosphere. Advances in the chemistry of UV systems, as well as improvements in UV-curing equipment, have largely overcome the effects of this inhibition, and most UV curing is conducted in air. However, the chemistry used to mitigate oxygen inhibition in UV is not effective in EB curing. The EB cure of inks and coatings in the presence of oxygen also generates ozone, thus EB cure almost always is performed in an inert atmosphere, typically nitrogen.

UV curing is dependent upon sufficient light energy penetrating throughout the ink or coating to insure adequate polymerization at the bottom layer of the film. Materials like pigments, which absorb the UV light energy required for polymerization, reduce penetration of the light energy and subsequently limit the thickness at which the ink or coating can be cured. While the UV cure of a clear coating several hundred

microns thick is feasible, UV cure of a highly pigmented ink or coating is limited to relatively low film thickness.

In EB curing, the opacity of the coating is irrelevant. The energy potential (accelerating voltage) of the electrons and the density of the coating govern the depth to which a coating can be adequately polymerized. Most EB curing units are capable of generating accelerating voltages of 150 KeV or more. In most cases, 150 kilovolt electrons can easily penetrate films opaque to UV.

EB-curing equipment is a source of ionizing radiation. When accelerated electrons strike metal, X-rays are generated. Suitable shielding is required to prevent exposure to these X-rays. EB equipment used to cure inks and coatings contains thin lead shielding that lines the accelerating and curing chambers. This shielding allows personnel to work in close proximity to the operating EB equipment.

It must be noted that with UV equipment, direct exposure to the UV lights must be avoided. This is relatively easily accomplished with engineering controls. Also, in UV curing, residual photoinitiator fragments can impart odor and migrating species or extractables to the cured ink or coating, and increase yellowing with exposure to sunlight. However, there are specialty photoinitiators that can address these potential shortcomings. EB curing avoids these issues since photoinitiators are not required.

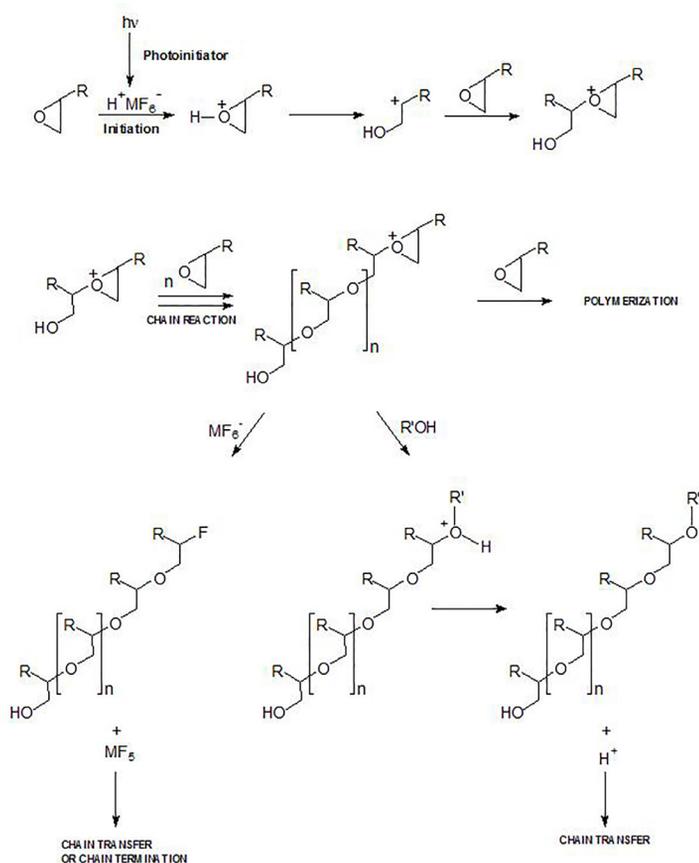
High EB doses can have a negative effect on some plastic and paper substrates, reducing strength or causing embrittlement. UV curing generally does not pose this problem.

Finally, the relative complexity of EB-curing units results in significantly higher equipment cost, roughly three times that for a typical UV installation.¹⁰

UV/EB cationic curing

While free radical curing chemistry accounts for a large majority of UV/EB curable formulations, another significantly utilized chemistry is the UV/EB-initiated cationic polymerization of epoxide functional materials.

In UV/EB cationic curing, a reactive chemical species is generated by the disassociation of a salt molecule (typically an arylsulfonium or aryl iodonium salt molecule) on exposure to UV or EB energy.¹¹ This reactive species opens the epoxy ring structure causing polymerization. Figure 9 shows the polymerization process for UV cationic cure systems. The steps of this process are analogous to those of the free radical process – acid formation (instead of radical formation), initiation, chain reaction (or propagation), chain transfer and termination. For EB cationic cure polymerization, an initiator is necessary, and acid is formed by interaction of the initiator with the electrons.¹²


FIGURE 9. UV cationic polymerization process

Cycloaliphatic epoxides are the most commonly used formulation component due to their higher reactivity (compared to glycidyl ether epoxies) and their relatively low viscosity. Hydroxy functional materials can co-polymerize with the epoxides, and polyols are commonly used to modify coating properties. Vinyl ether monomers also react cationically. Sometimes they are used as reactive diluents. Vinyl ether oligomers are also available.

UV/EB cationic curing systems can offer some performance advantages over free radical curing systems. The adhesion of cationic systems is generally superior to free radical systems, particularly on such substrates as unprimed metal and plastics. UV/EB cationic curing systems (those that involve the ring opening reaction) have significantly lower shrinkage upon polymerization.¹³ Table 5 highlights some significant distinctions between UV/EB radical and cationic curing systems. ■

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Radical	Cationic
Wide variety of raw materials	More limited raw materials
Inhibited by oxygen	Not inhibited by oxygen
Not inhibited by high humidity	Inhibited by high humidity
Not inhibited by basic materials	Inhibited by basic materials
Full cure in seconds	Full cure in hours unless given thermal "bump"
Adhesion: less	Adhesion: greater
Shrinkage: greater	Shrinkage: less
Depth of cure: greater	Depth of cure: less
Initiator needed for EB cure: no	Initiator needed for EB cure: yes

TABLE 5. Comparison of radical and cationic curing systems

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